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# Patchouli Oil Isolation and Identification of Chemical Components Using GC-MS

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**Abstract**

This research aims to isolate oil from patchouli (*Pogostemon cablin* Benth) and identify the chemical components of patchouli oil using Gas Chromatography-Mass Spectrometry (GC-MS). Patchouli plant samples were obtained from Welulu Village, Kolaka Regency, Southeast Sulawesi Province. Fresh patchouli leaves are dried in the sun for four days to a constant weight. Patchouli leaves water content is 77.41% (w/w). Dry patchouli leaves are distilled using the water and steam method for two hours. Water and oil distillates are separated using a separating funnel. Patchouli oil obtained was dried using $Na_2SO_4$ anhydrous and yield 0.73% (w/w). The results of GC-MS analysis showed that patchouli oil contained 13 chemical components compounds, namely β-patchoulene (4.56%), cedr-8-ene (1.24%), trans-caryophyllene (7.96%), α-guaiene (18.61%), seychellene (5.70%), patchoulene (2.88%), eremophilene (1.33%), azulene (8.74%), δ-guaiene (18.90%), cyclohexanone (1.10%), globulol (1.88%), veridiflorol (4.39%), and alcohol patchouli (22.7%).

*Keywords: GC-MS, isolation, identification, isolation, patchouli oil.*

## INTRODUCTION

Patchouli plants produce essential oils known as patchouli oil. There are three types of patchouli plants that grow in Indonesia, namely patchouli Aceh (*Pogostemon cablin* Benth) with an oil content of 2.5-5%, Javanese patchouli or forest patchouli (*Pogostemon heyneanus* Benth), and patchouli soap (*Pogostemon hortensis* Backer) with an oil content 0.5-1.5% each (Herdiani, 2011). Patchouli oil is used in perfume, aromatherapy, incense, flavorings, cosmetics, medicinal, and pharmaceutical industries (Ramya et al., 2013). Patchouli oil is used as a binder or fixator and until now has not been replaced by other ingredients. Indonesia is the world's leading producer of patchouli oil. Patchouli oil won 85% of Indonesia's essential oil exports, with a volume of 1,200-1,500 tons/year. Patchouli oil is exported to Singapore, United States, Britain, Spain, France, Switzerland, Germany, The Netherlands, Hong Kong, Egypt, and Saudi Arabia (Ditjenbun, 2020).

Patchouli oil consists of a mixture of compounds of terpenes with alcohol, aldehydes, and esters, which give a distinctive odor. Patchouli alcohol is the main component and determined the odor of patchouli oil. Isolation of essential oils from plant parts (roots, stems, bark, leaves, flowers) is carried out by water and steam distillation methods. Patchouli oil can be isolated from patchouli leaves by the water and steam distillation method. The gas chromatography method determines the amount and relative percentage of the components of patchouli oil (Sastrohamidjojo, 2007).

Patchouli is an antiseptic and insecticide used in traditional Chinese, Malaysian, and Japanese medicine. Characteristics of the aroma of patchouli distinguish Indian ink. Patchouli scents and improves ink color, while patchouli alcohol helps the ink to dry faster. Patchouli contains aldehyde, ketone patchoulenone, sesquiterpenes, monoterpene pinene, azulene, and eugenol (ACHS, 2012). Idris et al. (2014), conducted an analysis of SNI for patchouli oil quality, including specific gravity, refractive index, amount of acid, ester number, and solubility in alcohol.

Several studies on the analysis of patchouli oil chemical components using GC-MS have been conducted. Harahap (2009), identified patchouli leaves oil from Aceh, obtained five main components, namely β-patchoulene (3.13%), diepi-α-cendrene (4.06%), δ-guiaene (4.36%), delta-guiaene (4.82%), and alcohol patchouli (62.59%). Aisyah (2010), identified patchouli leaves oil from South Aceh, obtained five main components, namely Alcohol patchouli (32.60%), δ-guaiene (23.07%), α-guaiene (15.91%), seychellene (6.95%), and α-patchoulene (5.47%). Permana (2012), identified patchouli leaves oil from West Java, obtained five main components, namely alcohol patchouli (31.5%), α-bulnesene (12.3%), α-guaiene (11.7%), α-patchoulene (5%), and α-selinene (3.9%).

Different environmental conditions in each region affect the chemical components of plant constituents (Munwar, 2011). Ramayana dan Widyawati (2013), tested the steam distillation method to isolate the essential oil components of patchouli plants. This method has the advantage of producing good quality oil from the leaves of the patchouli plant through the hydro diffusion process. Around 80% of the population of Kolaka Regency is engaged in the plantation sector (Tamrin et al., 2015). In recent years, people in Kolaka Regency have begun cultivating patchouli plants in addition to farming that has been occupied previously. As an effort to improve production quality in accordance with SNI for patchouli oil, a study was conducted on the isolation of patchouli oil from Kolaka Regency and the identification of chemical components using GC-MS as one of the determinants of patchouli oil quality.

## METHODOLOGY

### Materials and Instrumentals

The materials used are patchouli leaves from Welulu Village, Kolaka Regency, Southeast Sulawesi Province, $Na_2SO_4$ anhydrous (p.a. Merck), bi-distilled water, and filter paper. The instrumentals used are Gas Chromatography-Mass Spectrometers (GC-MS QP-2010 Plus, Shimadzu), a set of distillation equipment (stainless steel), laboratory glassware (Pyrex), hot plate (Cimarec 1), and analytical scales (Ohaus).

### Methods

Patchouli leaves samples were taken in Welulu Village, Kolaka Regency, Southeast Sulawesi Province. Patchouli plants at the age of harvest, the leaves are taken, sorted, cleaned, washed, and dried in the sun for four days to a constant weight, then the water content is calculated. 1.05 kg of dry patchouli leaves were put into the kettle and distilled for two hours. The resulting distillate forms two layers, the upper layer is patchouli oil, and the lower layer is water. The oil is separated using a separating funnel, then added anhydrous sodium sulfate ($Na_2SO_4$), filtered, and weighed as water-free oil. Patchouli oil is obtained analyzed using GC-MS to identify chemical components.

## RESULTS AND DISCUSSIONS

### Isolation Oil from Patchouli Leaves

Patchouli leaves water content is calculated based on the weight difference before and after drying. Patchouli leaves water content obtained 77.41% (w/w). According to Sumarsono (2005), patchouli leaves water content after drying is around 70%. The process of drying patchouli leaves serves to provide resistance to the leaves so they do not get attacked by fungi quickly due to an enzymatic process that can reduce oil yield. Drying the sample also speeds up the refining time by assisting in hydro diffusion, which is the process of the entry of steam into the plant cell walls. Water molecules contained in the leaf cell layer can be minimized so that the distillation process runs more efficiently (Abdjul et al., 2012).

Patchouli oil isolation from 1.05 kg of dried patchouli leaves was done by distilling water and steam for two hours. Patchouli oil is obtained as much as 0.73% with bright yellow color and the typical aroma of patchouli by Indonesian National Standards quality (SNI 06-2385-2006) of yellow to reddish-brown patchouli oil's color (BSN, 2019). Patchouli oil yield in this study is similar to the results of Risnawaty et al. (2017), which is 0.88% and Slamet et al. (2019), is 0.98%, but relatively low compared to other studies using the steam distillation method with pressure reduction and fermentation methods (Harimurti et al., 2012 and Muharam et al., 2017).

Patchouli plants in Indonesia are widely cultivated by farmers using random seeds, simple technology, and minimal production facilities. Therefore, both production and productivity and the quality of oil produced are still low. Harvesting and postharvest handling activities have not been done correctly and adequately, such as how to harvest, harvesting time, handling material collected before refining. The handling of the harvested plant material to be extracted is closely related to the quality and yield of patchouli oil produced (Ministry of Agriculture, 2012). According to Arpi et al. (2011), the patchouli raw material source and the distillation method used will affect the clarity of patchouli oil and patchouli alcohol content. The longer the distillation time, the higher the patchouli oil yield, and the higher the content of patchouli alcohol.

**Patchouli Oil Component Identification**

The identification of patchouli oil components using GC-MS obtained 13 peaks, as shown in Figure 1 and Table 1.

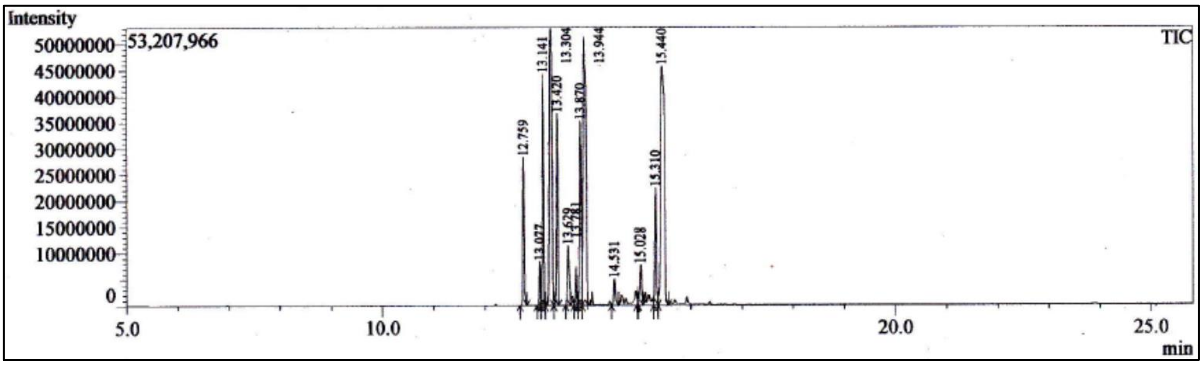


Figure 1. Patchouli oil chromatogram

Each peak is further identified by comparing the mass spectrum with reference compounds in the GC-MS database. Patchouli leaf oil component is determined based on SI (Similarity Index) value, which refers to compatibility with the compound fragmentation pattern.

Peak 1, with a retention time of 12.759 minutes, is β-patchoulene ($M^{+\cdot}$, m/z=204). Peak 2 with a retention time of 13.077 minutes is Cedr-8-ene ($M^{+\cdot}$, m/z=204). Peak 3, with a retention time of 13,141 minutes, is trans-caryophyllene ($M^{+\cdot}$, m/z=204). Peak 4, with a retention time of 13.304 minutes, is α-guaiene ($M^{+\cdot}$, m/z=204). Peak 5, with a retention time of 13.420 minutes, is seychellene ($M^{+\cdot}$, m/z=204). Peak 6, with a retention time of 13.629 minutes, is patchoulene ($M^{+\cdot}$, m/z=204). Peak 7, with a retention time of 13.742 minutes, is eremophylene ($M^{+\cdot}$, m/z=204). Peak 8, with a retention time of 13.870 minutes, is azulene ($M^{+\cdot}$, m/z=204). Peak 9, with a retention time of 13.944 minutes, is δ-guaiene ($M^{+\cdot}$, m/z=204). Peak 10, with a retention time of 14.531 minutes, is cyclohexanone ($M^{+\cdot}$, m/z=206). Peak 11, with a retention time of 15.028 minutes, is globulol ($M^{+\cdot}$, m/z=222). Peak 12, with a retention time of 15.310 minutes, is veridiflorol ($M^{+\cdot}$, m/z=204). Peak 13, with a retention time of 15.440 minutes, is alcohol alcohol ($M^{+\cdot}$, m/z=222).

Table 1. Patchouli oil components

| Peak | Area% | Component name |
|---|---|---|
| 1 | 4.56 | β-patchoulene |
| 2 | 1.24 | Cedr-8-ene |
| 3 | 7.96 | trans-caryophyllene |
| 4 | 18.61 | α-guaiene |
| 5 | 5.70 | Seychellene |
| 6 | 2.88 | Patchoulene |
| 7 | 1.33 | Eremophilene |
| 8 | 8.74 | Azulene |
| 9 | 18.90 | δ-guaiene |
| 10 | 1.10 | Cyclohexanone |
| 11 | 1.88 | Globulol |
| 12 | 4.39 | Veridiflorol |
| 13 | 22.70 | Alcohol patchouli |

The three main components of patchouli oil are α-guaiene (18.61%), δ-guaiena (18.90%), and alcohol patchouli (22.70%). The results of this study are almost the same as Abdjul et al. (2012), that identified patchouli leaves oil from Malang, obtained three main components, namely alcohol patchouli (20.36%), δ-guaiene (14.50%), and α-guaiene (12.89%). Differences in the composition of patchouli oil are qualitatively and quantitatively due to differences in environmental factors, the area of origin of the sample, differences in harvesting methods, postharvest processing, refining conditions, and oil storage (Aisyah, 2010).

The α-guaiene mass spectrum (m/z): 30, 41, 55, 67, 79, 93, 105, 119, 133, 147 $[C_{15}H_{24}]^{+\cdot}$ (base peak), 161, 175, 189, and 204 as shown in Figure 2. The structure of α-guaiene is shown in Figure 3.

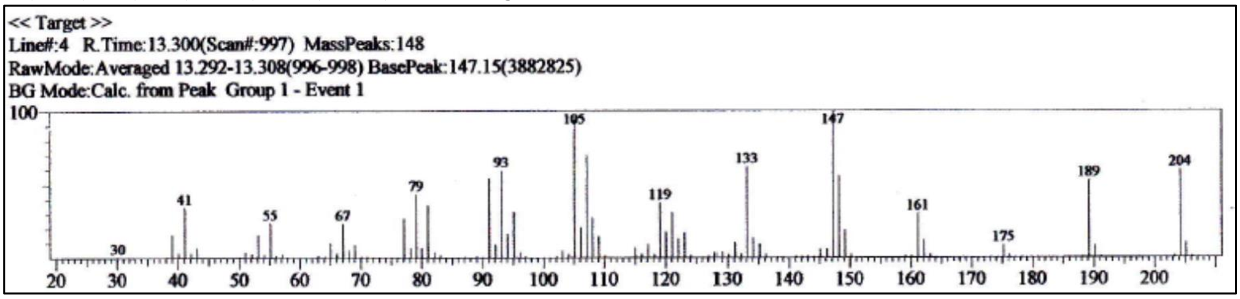


Figure 2. α-guaiene mass spectrum

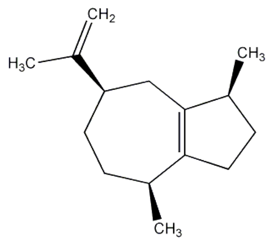

Figure 3. α-guaiene

The δ-guaiene mass spectrum (m/z): 30, 41, 55, 67, 79, 93, 107 $[C_{15}H_{24}]^{+\cdot}$ (base peak), 119, 133, 148, 161, 175, 189, and 204 as shown in Figure 4. The structure of δ-guaiene is shown in Figure 5.

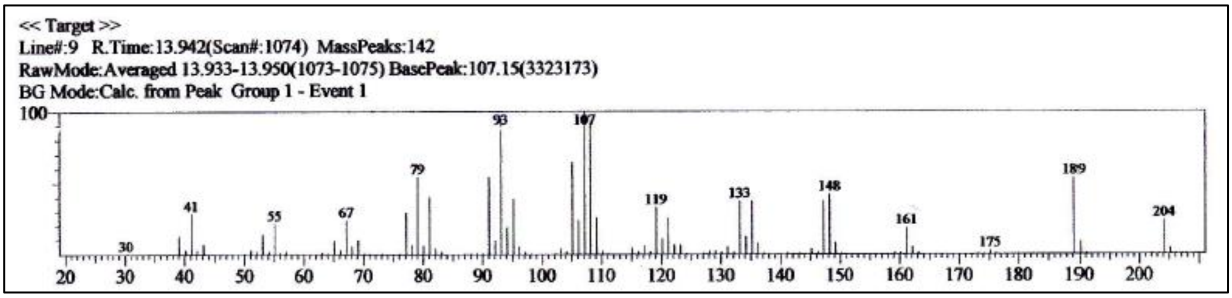


Figure 4. δ-guaiene mass spectrum

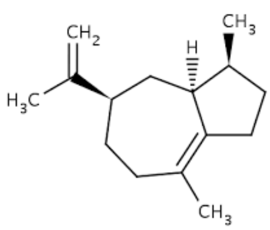

Figure 5. δ-guaiene

The guaienes are used in the fragrance and flavoring industries to impart spicy aromas and tastes. The α-guaiene and δ-guaiene compounds in the industry are usually used as room fresheners (Abdjul et al., 2012).

The alcohol patchouli mass spectrum (m/z): 30, 41, 55, 67, 83 $[C_{15}H_{26}O]^{+\cdot}$ (base peak), 98, 125, 138, 151, 161, 179, 189, 207, and 222 as in shown Figure 6.

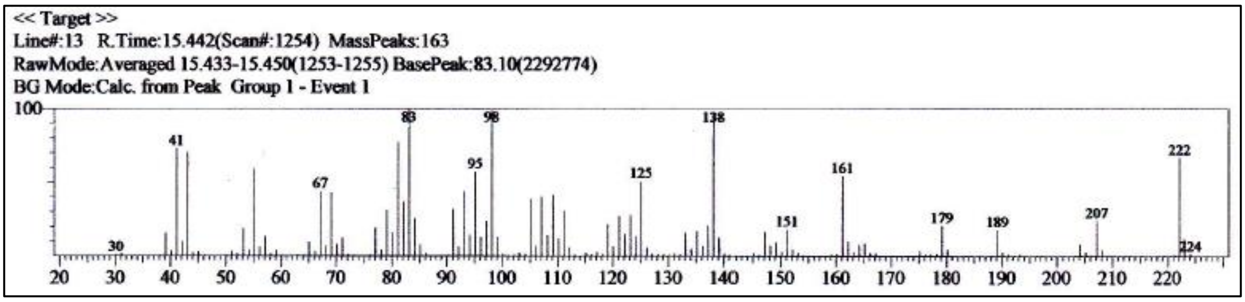


Figure 6. Alcohol patchouli mass spectrum

The structure of alcohol patchouli is shown in Figure 7.

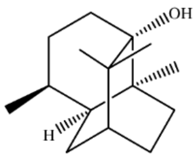


Figure 7. Alcohol patchouli

Alcohol patchouli level is one of the parameters that determine the quality of patchouli oil. Alcohol patchouli has a high selling value because it can bind the scent, so it is widely used in the perfume industry Ermaya et al. (2019). The national standard for the best quality of patchouli oil is at least 31% alcohol patchouli (BSN, 2019). Patchouli oil produced in Indonesia has an alcohol patchouli level of <30% because the handling of post-harvest material before refining is not proper; the distillation process is not optimal with a short distillation time, and the influence of the origin of the raw material. Therefore alcohol patchouli content still needs to be increased to expand its market reach (Aisyah et al., 2010).

## CONCLUSIONS

Patchouli leaves oil isolation obtained 0.73% yield. The results of GC-MS analysis showed that patchouli oil contained 13 chemical components compounds, namely β-patchoulene (4.56%), cedr-8-ene (1.24%), trans-caryophyllene (7.96%), α-guaiene (18.61%), seychellene (5.70%), patchoulene (2.88%), eremophilene (1.33%), azulene (8.74%), δ-guaiene (18.90%), cyclohexanone (1.10%), globulol (1.88%), veridiflorol (4.39%), and alcohol patchouli (22.7%).

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